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Tensile Strength of Poly-*p*- Phenylene Benzobisoxazole (PBO) Fiber with Kinking Damage

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Abstract

In this study, tensile strength of PBO fiber with kink band was investigated in single fiber (monofilament) tensile tests. The kink band was introduced by the wrapping fiber bundle to the steel bars with several diameters. Weibull analysis on the obtained tensile strength was carried out to discuss the strength in the on region of kink band. It was found that the tensile strength of PBO fiber decreases with the increase in kink band density. Kink bands act as defects that degrade the tensile strength. A Weibull analysis demonstrated that the concept of effective volume explains the tensile strength of PBO fiber. The reduction of tensile strength at low bar diameters from the appearance of kink bands is not due to changes in strength near the kink bands but rather to the increase in kink band density.

Keywords: PBO fiber, Kink band, Tensile strength, Weibull analysis

1. Introduction

Poly-*p*-phenylene benzobisoxazole (PBO) fiber is an aromatic, heterocyclic fiber spun from highly oriented PBO

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chains, which are quite tough and have a straight structure. This fiber offers superior strength and elastic moduli with a tensile strength and tensile elastic modulus over double those of aramid fibers. Its thermal resistance and flame-resistance are also superior to other organic fibers, and it is believed to offer great potential as a replacement for conventional fibers (Yabuki (1995)). Like other high-molecular weight fibers, however, its compressive strength is far lower than its tensile strength, and microscopic alterations in its internal structure occur under bending load, with formation of what is called a kink band (Chau et al. (1995), Leal et al. (2009), Lorenzo-Villafranca et al. (2012)). Conventionally, since the kink band consists of a collection of buckled microfibrils, it has been believed that buckling is counteracted partially or completely and thus moderates the resulting reduction in tensile strength by stretching the microfibrils during tensile loading. This is why there have been very few published studies of the tensile and fatigue strengths of fibers in which the kink band has occurred. The strength of PBO fiber lends it to uses from optical cable to power cords for portable electronics (Furukawa (2006)), and the allowed curvature for the fiber has been improved to shorter and shorter radii every year, to very small radii nearly corresponding to bending fibers double. Inspection based on the experiment are needed to determine whether the kink band actually has only a minor effect on the fiber strength. Also, if quantitative data can be taken regarding how the tensile strength of PBO fiber is related to the kink band, these will be extremely useful findings, because they will indicate safe values for the tensile strength. In consideration of the above facts, the authors of this report have carried out a series of studies of the mechanical properties of PBO fiber (Horikawa et al. (2005, 2007, 2008, 2009)). Our previous report (Horikawa et al. (2013)) examined PBO fiber that contained the kink band and demonstrated how the tensile strength varied with kink band density. Next, PBO fiber was wrapped around steel bars of various diameters and a single-fiber tensile test was carried out on the fibers with the resulting kink bands. It was demonstrated that (1) the number of kink bands increased as the diameter of the wrapping bar for the PBO fiber decreased, that is, as the compressive strain at the fiber surface increased; (2) the presence of kink bands reduces the tensile strength, that is, kink bands permanently damage the tensile strength; and (3) the tensile strength decreases with the increase in kink band density. Nonetheless, it is still unknown whether changes in the strength following the appearance of kink bands are due to the compressive strain of the fibers, because no reports have addressed this matter. This study employs the strength data of PBO fiber measured from a Weibull analysis in the previous report (Horikawa et al. (2013)) to examine whether the strength of the portion with kink bands changes due to compressive strain.

2. Experimental Procedure

2.1. Test Material

PBO fiber (Zylon®-HM, Toyobo) was used in this experiment. Figure 1 shows the molecular structure of the PBO fiber. The molecular structure consists of linear aromatic rings. The molecules are connected in the direction of the fiber, and their consistent orientation leads to its high coefficient of elasticity. Table 1 shows the catalogued values for the mechanical properties of PBO fiber (Technical Information (Revised 2005.6)). As shown in the table, the compressive strength of the fiber is far lower than its tensile strength. Figure 2 provides micrographs of the exterior and the cross section of a PBO fiber. The fiber surface is relatively smooth and its cross section is nearly circular.

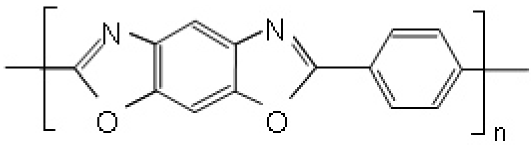


Fig. 1. Molecular structure of PBO fiber.

Table 1. Catalog values for mechanical properties of PBO fiber (Technical Information (Revised 2005.6)).

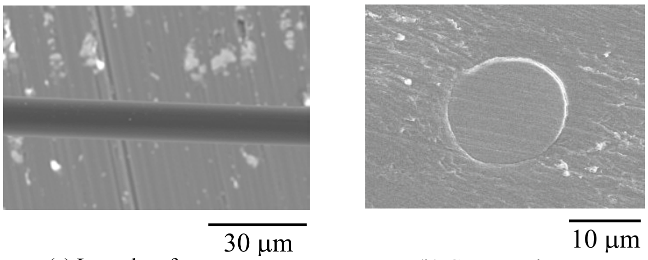
Tensile Elastic Modulus (GPa)	Tensile Strength (GPa)	Compressive Strength (GPa)
270	5.8	0.561

2.2. Method for introducing kink bands

The method for introducing kink bands into a PBO fiber was the same as in the previous report (Horikawa et al. (2013)), but is summarized here. The PBO fiber was supplied from the manufacturer in bundles of about 300 fibers. Kink bands were introduced by wrapping the bundles around steel bars. Figure 3 is a diagram of this process. One end of the bundle was fixed to the bar. To apply a constant tensile load to the bundle while wrapping it, a weight was hung from the other end of the bundle (corresponding to 0.1% of the tensile strength of the bundle). Wrapping was controlled to prevent overlapping on the bar. The bar diameter was calculated as follows. Each single fiber was assumed to be an isotropic elastic solid, and the tensile elastic modulus was assumed equal to the compressive elastic modulus given in Table 1. The compressive strain at failure was calculated by using the compressive strength and compressive elastic modulus. Next, the mean diameter, about 10.7μm, of the PBO fiber was used in Eq. (1) to estimate bar diameter at compressive failure during wrapping. The bar diameter, about 5mm, at compressive failure was set as the standard, and four bar diameters were used, 1×, 1/2×, 1/4×, and 1/8× the standard size, to introduce a kink band into a fiber bundle. Table 2 provides the conditions under which the kink bands were introduced.

$$\varepsilon \approx d_{fiber}/d_{steel\ bar}\ ,\quad d_{fiber} \ll d_{steel\ bar} \tag{1}$$

In Eq. (1), ε, d_{fiber}, and $d_{steel\ bar}$ represent the compressive strain at the surface of the fiber, the diameter of each fiber (the average of fiber diameter is only used when calculating the average of surface compressive strain in Table 2), and the steel bar diameter, respectively. A scanning electron microscope (SEM) was used to measure the fiber diameter. The fiber diameter was determined by the image analysis (WinROOF, MITANI Corporation) from the resulting SEM image. The fiber diameter in Table 2 are average of 40 specimens tested under each condition. An optical microscope (VHX-900, KEYENCE) was used to observe the kink bands forming in the fibers. Figure 4 is an optical photomicrograph of kink bands that formed on the surface of a fiber in a bundle wrapped around a bar. The kink



(a) Lateral surface. (b) Cross-section.

Fig. 2. Micrographs of PBO fiber.

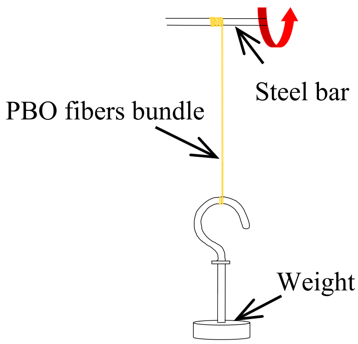


Fig. 3. Diagram of system for introducing a kink band.

Table 2. Conditions for introduction of kink band.

Diameter of steel bar (mm)	5	2.5	1.25	0.65
Average of fiber diameter (μm)	10.3	10.5	10.6	10.6
Average of surface compressive strain on the wrapped fiber (%)	-0.21	-0.42	-0.85	-1.63

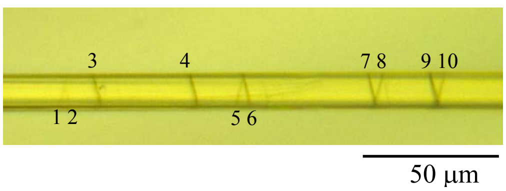


Fig. 4. Example of optical photomicrograph of kink bands.

bands, shown as black strips in this figure, confirm their formation during wrapping. In the previous study (Horikawa et al. (2013)), the authors defined the kink band density as the number of kink bands found per unit length of individual fiber. This study presents a quantitative evaluation of the relation between the value of the density and the tensile strength. The number of kink bands per fiber length of 250 μm was counted under the optical microscope and converted to a number per 100 μm to define the kink band density. As shown by the numerals in the figure, the number of kink bands was counted by assuming a black strip as a single defect. The bands in each test specimen were counted outside the gauge region (A in Fig. 5.) before the tensile tests.

2.3. Specimen shape

Because PBO fiber is extremely fine, paper tabs as defined in JIS R 7606 (gauge length 12.5 mm) were used for the tensile test of single fibers. Figure 5 is a diagram of a single fiber specimen. A single fiber was removed from the bundle, wrapped around the bar, and fixed at each end to the paper tabs with epoxy adhesive to create a specimen. Since PBO fiber loses strength under exposure to visible light, the specimens were stored in the dark until the time of the tensile test.

2.4. Single fiber tensile test (Monofilament Tensile Test)

A small table-top tester (SHIMADZU CORPORATION, EZ-Test) was used for the tensile tests of single fibers. For the test, the paper tabs attached to a fiber were fixed in the chucks of the tester and cut with scissors at each end, and the load was applied. The load was measured with a 5 N load cell, and the cross-head motion was measured simultaneously. The tension rate was set at 0.5 mm/min and the number of specimens tested under each condition was set at 40, in anticipation of scatter in the strengths. There is also scatter in the diameters of PBO fibers, so the diameter of each specimen was measured on a SEM image and used to calculate the tensile strength of that specimen. The diameters were measured with the SEM after completion of the tensile test in order to avoid deformation and defects due to heating of the fiber with the electron beam. These measurements were carried out in portions of the fibers outside the gauge region, and thus not subject to the actual tensile forces (A in Fig. 5). Fibers in which the failure occurred at the bonded locations rather than within the gauge region were eliminated from the dataset.

3. Experimental Results and Discussion

3.1. Tensile strength of PBO fiber containing kink bands

Figure 6 is a plot of the Weibull probability of the tensile strengths of PBO fibers incorporating kink bands at all rod diameters. The strengths of the relatively weak PBO fibers incorporating kink bands form a curve on the graph, but the overall pattern is linear. This result suggests that the bands comply with a two-parameter Weibull distribution. Table 3 lists the shape parameters and the scale parameters. The shape parameters for the diameter of steel bar 0.65-

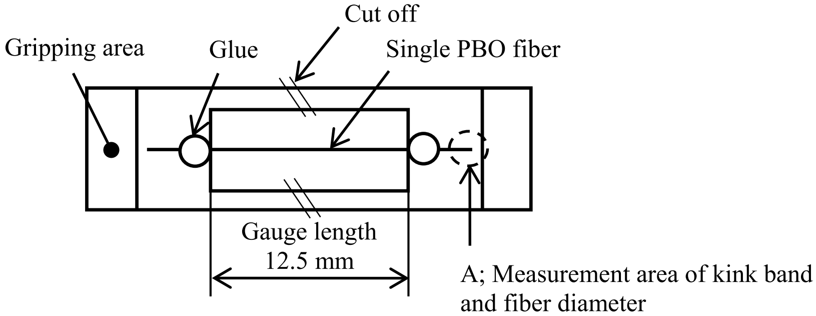


Fig. 5. Tensile specimen of single PBO fiber.

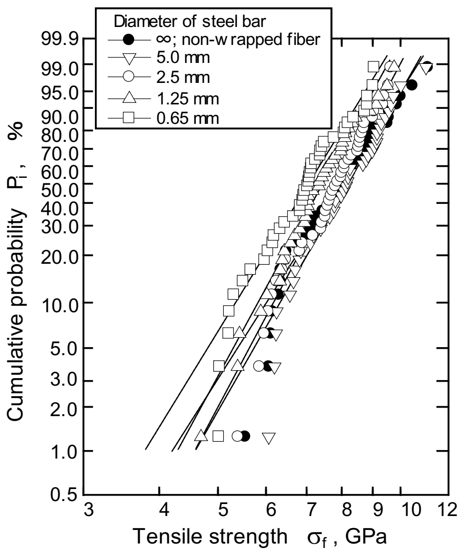


Fig. 6. Distribution of strengths of PBO fiber incorporating kink bands (Weibull probability graph)

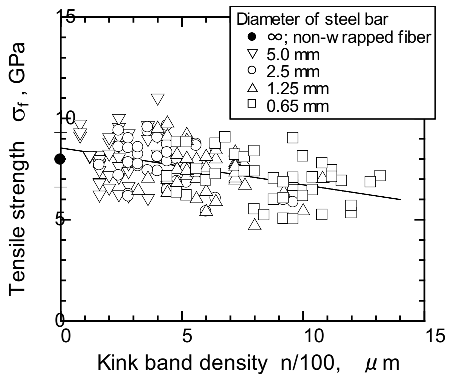


Fig. 7. Variation in tensile strength with kink band density (Horikawa et al. (2013)).

Table 3. Weibull parameter values for PBO fibers incorporating kink bands.

Diameter of steel bar (mm)	Shape parameter	Scale parameter
∞	6.56	8.47
5.0	7.29	8.62
2.5	7.85	8.22
1.25	7.67	7.80
0.65	6.80	7.44

2.5 mm decreases with decreasing the bar diameter, that is, with increasing compressive strain on surface of the fiber. The scale parameter, which is an index of the strength, shows a tendency to diminish with decreasing bar diameter. Figure 7 shows the relation between tensile strength and kink band density found in the previous report (Horikawa et al. (2013)). It is found that the tensile strength decreases with an increase in the number of kink bands. The data provided in Figs. 6 and 7 are useful because they show the relations of the tensile strength with both the bar diameter and the kink band density; however, as Fig. 8 shows, the tensile strength of PBO fiber decreases with an increase in the fiber diameter, so there is also a size effect in the diameter direction. Since the results in Figs. 6 and 7 came from PBO fibers of differing diameters, they include the size effect in the diameter direction. Also, the results in Figs. 6 and 7 indicate a tendency for the tensile strength to diminish with decreasing bar diameter, but it is unclear whether this was caused by an increase in the number of kink bands or by a decrease in strength of the locations where the kink bands appeared. Considering the above, the authors defined the residual strength ratio R as the ratio between $\sigma_{f,non\text{-}kink}$, the tensile strength of fibers in which a kink band did not occur, and $\sigma_{f,kink}$, the tensile strength of fibers in which a kink band did occur. The residual strength ratio R is expressed by the following Eq. (2).

$$R = \sigma_{f,kink}/\sigma_{f,non-kink} \quad (2)$$

The authors propose R as a strength parameter for PBO fiber (Horikawa et al. (2013)). The straight line in Fig. 8 is used to estimate $\sigma_{f,non\text{-}kink}$, for each fiber that developed kink bands. Figure 9 shows how the residual strength varied with kink band density. The figures show the residual strength ratio decreasing with the increase in kink band density. In our previous report (Horikawa et al. (2013)), the authors demonstrated that this decrease in strength originated not from differences in the diameters of the fibers but from defects caused by the kink bands.

3.2. Weibull analysis of tensile strength of PBO fiber incorporating kink bands

From the results in Fig. 9, it is clear that the residual strength ratio of the fibers, that is, the strength of the fibers, decreases with an increase in kink band density. Furthermore, the presence of R = 1 or more of the data in Fig. 9 are not increased strength by introducing kink bands. It is considered to be due to the variation in data points in Fig. 8. However, it is still unclear whether the strength at the kink bands has anything to do with the compressive strain imposed by the kink bands. A Weibull analysis of fibers possessing identical kink band densities must be performed to answer this question. Therefore, an analysis was carried out on the source data for Fig. 9 to see whether the strength at the kink bands varies due to the compressive strain imposed on the fibers. First, the strengths found for various bar diameters were classified by kink band density. It was assumed that the kink bands showed constant distributions in the fiber direction, and the Weibull analysis was performed. The following equation for a two-parameter Weibull distribution was assumed to apply to the residual strength ratio of fibers that had identical bar diameters and contained kink bands:

$$\mathrm{P}(R) = 1 - exp\left\{-V_e\left(\frac{R}{R_b}\right)^{m_R}\right\} \tag{3}$$

where V_e, R_b, and m_R are the effective volume, a scale parameter, and a shape parameter, respectively. The mean residual strength ratio $\overline{R}$ was calculated as

$$\overline{R} = R_b \cdot V_e^{-\frac{1}{m_R}} \cdot \Gamma\left(1 + \frac{1}{m_R}\right) \tag{4}$$

where Γ is the gamma function. The mean residual strength ratio for the specimens of differing shape and dimensions was calculated by using a relational equation expressing the volume effect, derived from Eq. (4):

$$\frac{\overline{R_2}}{\overline{R_1}} = \left(\frac{V_{e2}}{V_{e1}}\right)^{-\frac{1}{m_R}} \tag{5}$$

where $\overline{R_1}$ and $\overline{R_2}$ represent the mean residual strengths, and V_{e1} and V_{e2} represent the effective volumes. If the kink bands forming in the fiber are uniformly distributed throughout the surface and failure occurs due to the kink bands, the effective volume from Eq. (5) can be interpreted as the kink band density per unit area. Thus, if the kink band densities when the mean residual strengths are $\overline{R_1}$ and $\overline{R_2}$ are designated as D_1 and D_2, Eq. (5) can be re-written as

$$\frac{\overline{R_2}}{\overline{R_1}} = \left(\frac{D_2}{D_1}\right)^{-\frac{1}{m_D}}, \quad m_R = m_D \tag{6}$$

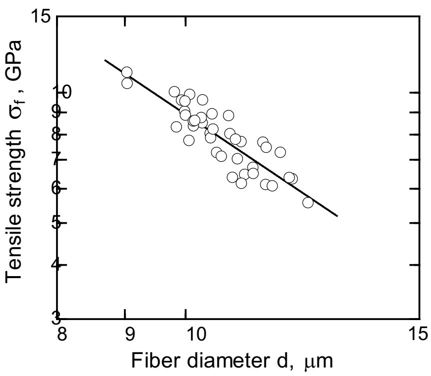


Fig. 8. Variation in tensile strength with fiber diameter for PBO fiber in which a kink band does not occur (Horikawa et al. (2013)).

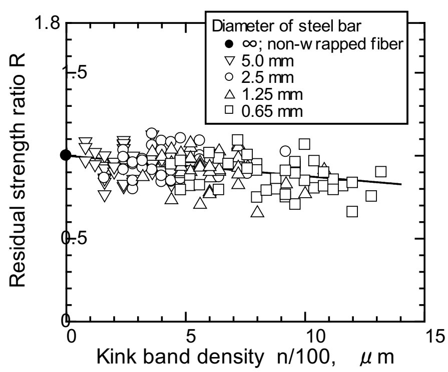


Fig. 9. Variation in residual strength with kink band density.

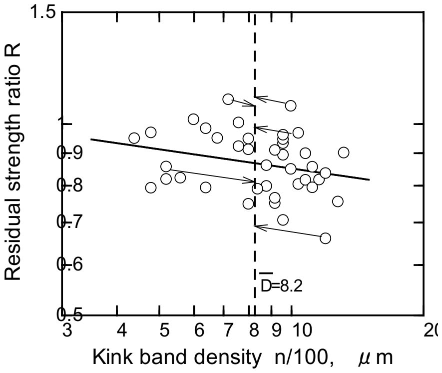


Fig. 10. Variation in residual strength with kink band density (bar diameter 0.65 mm)

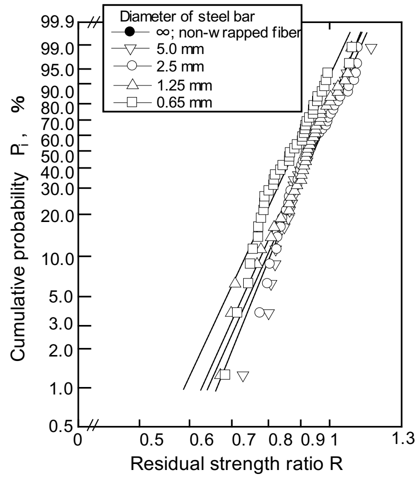


Fig. 11. Distribution of residual strengths of PBO fiber incorporating kink bands (Weibull probability graph)

Table 4. Weibull parameter values for residual strength of PBO fiber.

Diameter of steel bar (mm)	Shape parameter m_R	Scale parameter R_b
∞	—	—
5.0	12.12	0.97
2.5	10.96	0.98
1.25	10.88	0.96
0.65	10.51	0.91

where m_D is the shape parameter for the Weibull distribution describing the kink band density. Equation (6) in turn can be re-written as

$$\log \overline{R} = -\frac{1}{m_D} \cdot \log D + C_D \quad , (C_D \text{ is a constant}). \tag{7}$$

Figure 10 shows the relation between the residual strength ratio and the kink band density as a log-log plot when the bar diameter was 0.65 mm as an example. The vertical axis is residual strength ratio and the horizontal axis is kink band density. The solid line is a least-squares approximation. Here, to calculate the residual strength ratio corresponding to any mean kink band density at any bar diameter, the corresponding points on the diagram are moved parallel to the appropriate kink band density along the $-1/m_D$ gradient line (arrow in Fig. 10). However, there is scatter in the kink band density away from the residual strength ratio, and because no residual strength ratio obtained at constant kink band density, m_D is strictly unknown. That is why the slope of the regression line in the figure was set to −1/mD in Eq. (7). To be more specific, the data points were moved parallel to the solid line in the figure, and the residual strength ratio was converted to a value that corresponds to the mean kink band density. This operation was carried out for every group of bar diameters to find all of the residual strength ratio values.

Figure 11 is a Weibull probability plot of the residual fiber strengths corresponding to mean kink band density values at various bar diameters. It is found that residual strength ratio comply with the two-parameter Weibull distribution because the residual strength ratio is approximated by a straight line on Weibull probability graph. Table 4 presents the scale and shape parameters for each bar diameter. Figure 12 presents variation in the scale parameter obtained in the Weibull analysis with the kink band density. Figure 12 shows the tendency for the scale parameter, which has some consistent values, to slope off to the right at bar diameters of 2.5, 1.25, and 0.65 mm. If this portion is approximated with the least-squares method, the slope is 0.1218. Applying this value to Eq. (7), we find a value for m_D of 8.21, which is close to the mean value of 10.78 for the shape parameters in Table 4 for the bar diameters of 2.5, 1.25, and 0.65 mm. This shows that at the kink band densities in these bar diameters, the tensile strength of PBO fiber containing kink bands is explained by the concept of effective volume. The decrease in fiber tensile strength due to

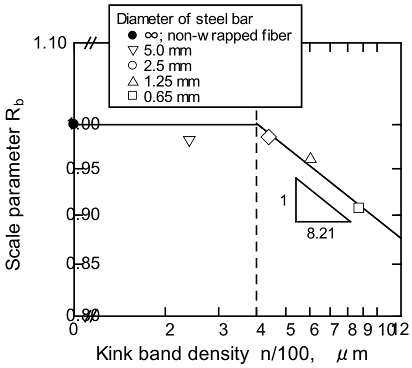


Fig. 12. Variation in scale parameter obtained by the Weibull nalysis with kink band density.

the occurrence of kink bands is not particularly due to changes in the strength of the region where a kink band forms, that is, the strength value of the region where a kink band forms is constant, even at small bar diameters. Rather, the decrease in fiber tensile strength can be attributed to the increase in the density of kink bands. The scale parameter for the bar diameter of 5.0 mm is nearly the same as that for the bar diameter of 2.5 mm. It could be that the assumption of the Weibull analysis of uniform distribution in the kink bands does not hold when the bar diameter is large, i.e., when there is excessive scatter in the spacing of the kink bands forming in the fiber, due to low compressive strain at the fiber surface.

4. Summary

(1) The tensile strength of PBO fiber decreases with the increase in kink band density. Kink bands act as defects that degrade the tensile strength.

(2) A Weibull analysis demonstrated that the concept of effective volume explains the tensile strength of PBO fiber. The reduction of tensile strength at low bar diameters from the appearance of kink bands is not due to changes in strength near the kink bands but rather to the increase in kink band density.

Acknowledgements

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TOYOBO CO., LTD., ZYLON® (PBO fiber) Technical Information (Revised 2005.6)